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(54) Encapsulated liquid detergent composition.

(57) Encapsulated, essentially non-aqueous liquid detergent composition, wherein the liquid detergent component comprises a non-aqueous organic solvent and particles of a solid material dispersed therein, encapsulated in a water-dispersible substance. Preferably, the solid material comprises a builder.

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## Encapsulated Liquid Detergent Composition

The present invention relates to an encapsulated, essentially non-aqueous liquid detergent composition.

In recent years, much effort has been put into developing essentially non-aqueous liquid detergent compositions. Such compositions are dispersions comprising a non-aqueous liquid (solvent) phase which can be a liquid surfactant, an organic non-aqueous non-surfactant liquid or a mixture of such materials.

5 Furthermore, they contain dispersed particulate solids. These are small (e.g. 10 microns) particles of solid material which are useful in cleaning, such as solid surfactants, builders, bleaches, enzymes or any other of such solids known in the art.

10 Although such non-aqueous liquid detergent compositions are advantageous in that they constitute a very concentrated product form, the stability of the liquids leaves much to be desired. Furthermore, their handling and dispensing properties have proven to be problematic. A product having an optimal viscosity is difficult to formulate because of conflicting requirements. If the viscosity of the liquid product is too high, substantial amounts thereof may remain in the dispenser of the washing machine. If, on the other hand, its viscosity is too low the liquid detergent may leak down the drain of the European type washing machines. Furthermore upon contact with water the non-aqueous liquids tend to form gels, which again causes 15 dispensing problems. Moreover, as with all liquid detergent compositions, non-aqueous liquid detergent compositions are difficult to dose from the point of view of administering the right amount of detergent as well as the messiness during handling of the liquid products. Therefore it would be desirable to provide a suitable unit dosing form for these kind of compositions.

15 In order to obviate some of these problems, it has been proposed to encapsulate liquid detergent compositions in a self-dissolving capsule. The capsule, which is usually made of gelatin, imposes considerable restrictions on the type of liquid detergent to be used therein, and the encapsulation has only been successful in a few occasions. In particular, only essentially water-free or non-aqueous liquid detergent compositions can be used, as water tends to dissolve the gelatin capsule.

20 GB-A-2,192,404 (Scherer) relates to a foam bath product comprising 40-60% of amine salts of long chain alkyl ether sulphates, 25-35% low molecular weight polyethylene glycol and 2.5-7.5% glycerin encapsulated in soft elastic water-soluble gelatin.

25 The US-patent 3,528,925 (Chapuis) discloses an encapsulated synthetic liquid detergent composition consisting of 33-65% of an anionic surface active ingredient, 8-36.5% of a nonionic surface active ingredient and 20-59% of an alkanolamine or alkylamine. The composition is therefore essentially a non-built light duty 30 detergent composition. It contains less than 1% water and is encapsulated in gelatin.

35 The wash performance of the encapsulated liquid detergent compositions such as described in said US-patent is unsatisfactory. The necessity of using essentially water-free detergent compositions in view of the stability of the gelatine capsule evidently constitutes a serious limitation to the detergent composition at the expense of its detergency properties. For example, there are restrictions as to the pH of the content of the capsule. The US-patent 3,528,925 teaches the need of using a weak organic base, such as alkyl- or alkanolamines, in order to keep the pH below 8, although it is known that this pH is too low for an optimal laundry process. Furthermore, there is no builder present, probably to avoid unfavorable interactions with the other components, as well as stability- and or viscosity problems.

40 One object of the present invention is to provide an encapsulated, heavy duty liquid detergent composition. Another object is to provide an essentially non-aqueous liquid detergent composition, suitable for use in such encapsulated detergent product.

We have now found that these and other objectives can be achieved by the essentially non-aqueous encapsulated liquid detergent compositions according to the present invention.

45 In accordance with the first aspect of the present invention there is provided an encapsulated essentially non-aqueous liquid detergent composition, which is characterized in that it comprises a non-aqueous organic solvent and particles of a solid material dispersed therein, encapsulated in a water dispersible substance.

As liquid detergent compositions, preferably the liquid cleaning products are used which are described in the co-pending European patent application 87.309568.1 which is incorporated herein by reference.

50 This European patent application relates to non-aqueous liquid cleaning products which are formulated by dispersing particulate solids in an organic solvent by using a structurant or deflocculant which causes a viscosity reduction at low shear rates in that solids:solvent system or an equivalent system in which the solids volume fraction is sufficiently high to raise the viscosity of the solvent. The organic solvent may be a liquid surfactant. The solids are particles of a solid material which is useful in cleaning, such as surfactants, builders, bleaches, enzymes, etc. The structurant is any acid, salt or base which fulfills the aforementioned

test.

According to the present invention, the solid material which dispersed in the organic solvent is preferably a builder such as a zeolite or sodium tripolyphosphate (STP).

The encapsulating material is a water-dispersible substance. Preferably it comprises a water-soluble substance, e.g. gelatin, polyvinyl alcohol (PVA) or polyethylene glycol. Especially preferred as encapsulating material is gelatin. In this case it was surprisingly found that the liquid detergent composition inside the capsule may additionally contain one or more proteolytic enzymes, although it would be expected that these would digest the gelatine capsule.

The essentially non-aqueous liquid detergent compositions can be prepared as described in the above mentioned European patent application. They can be encapsulated in accordance with methods which are known to the man skilled in the art. For instance, the US-patent 3.528.925 describes the encapsulation in gelatin of a non-aqueous liquid detergent composition by means of two continuous ribbons of a melted gelatin mass.

The invention will now be further illustrated by the following examples.

15

#### Examples 1 - 2

20 The following non-aqueous detergent compositions (NAL'S) were used for encapsulation (the amounts are given as % by weight):

25

	1	2
Plurafac RA 30	29.7	30.0
Marlon	3	3
Glycerol triacetate	5	5
STP. Oaq	51.1	51.7
Soda ash	10.2	10.3
Savinase	1.0	--

30

These compositions were encapsulated into soft gelatin capsules by means of the rotary die process. In this known process two continuous ribbons of gelatin are formed and subsequently pressed together between two rotating drums having dies on their surfaces, whereby the liquid is added between the ribbons. Round capsules were produced containing approximately 7.5g liquid detergent. The properties of these capsules were evaluated with regard to release, storage stability and performance.

#### A. Release

40

The release rates and total delivery of the capsules were measured in a modified AEG drum-type washing machine, of which the front walls of both inner and outer drum were replaced by transparent plastic plates. The release of the capsule contents was registered by means of on-line conductivity in a 40 °C heat-up cycle with a water intake of 15 l of tap water and a wash load of 1 kg of mixed cotton. In all experiments, a number of capsules was dosed corresponding to approximately 30 g of NAL. The possible occurrence of mechanical loss was checked by recirculating the suds via the machine drain at the end of each experiment.

45

For all capsules, the time required to reach equilibrium concentration in the washing machine ranged between 8 and 9 minutes. The average temperature at which the initial release of NAL was observed, was equal to 26 °C, while the average temperature at the time of complete release was 33 °C. These results were obtained at a water-intake temperature of 19 °C and a heating rate of 1.4-1.5 °C/min.

50

In the experimental AEG machine, the percentage of mechanical loss was less than 10 % in the majority of all measurements.

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#### B. Storage stability

Spherical capsules containing non-aqueous liquid including 1 % savinase were stored under various conditions. The samples which were stored in closed cups at 37 °C showed a remaining activity after four

weeks of 75 %, which figure is comparable with normal proteolytic enzyme stability in current non-aqueous liquids. No degradation of the polypeptide gelatine capsule wall was observed under these conditions.

5    C. Performance

In an appraisal test, the performance of the encapsulated NAL was compared with the performance of standard NAL on various test cloths. The test was carried out in a top loading drum-type washing machine, main wash only (40 °C, 30 min), a water hardness of 15° FH and 3 kg of standard soiled load. The 10 performance of six spherical capsules (correponding to 45 g of NAL), with and without Savinase, was compared to the performance of 45 g non-encapsulated NAL, without savinase, as a control. The products were dosed in the drum between the load. The results of the test are shown in Table 1

Table 1

Single wash performance on testcloths (ΔR <sup>460</sup> )				
Example	1	2	Control	
Testcloths:				
AS09	24.4	23.9	19.9	
PC09	17.4	17.0	15.2	
WFK10c	10.3	10.6	8.5	
EMPA101	15.9	14.2	11.7	
BC-1	-1.1	-1.9	-2.0	
AS10	28.3	6.0	5.7	
PC10	22.3	5.8	5.5	
EMPA116	21.1	4.7	4.4	
Terry Towel	-2.0	-3.2	-3.2	

The experiments show that the encapsulated NAL'S, with and without the proteolytic enzyme, have very satisfying wash performance as measured by the delta-R values. Surprisingly, it was found that the performance of the NAL actually has been improved by the encapsulation in gelatine. These findings 35 suggest the occurrence of mechanical loss for the non-encapsulated liquids, resulting in a performance decrease.

40    **Claims**

1. Encapsulated, essentially non-aqueous liquid detergent composition, wherein the liquid detergent component comprises a non-aqueous organic solvent and particles of a solid material dispersed therein, encapsulated in a water-dispersible substance.
2. Encapsulated detergent composition according to claim 1, wherein the dispersed solid material comprises a builder.
3. Encapsulated detergent composition according to claim 2, wherein the builder is sodium tripolyphosphate.
4. Encapsulated detergent composition according to any of claims 1-3, wherein the liquid detergent composition contains one or more enzymes.
5. Encapsulated detergent composition according to any of claims 1-4, wherein the water-dispersible substance comprises a water-soluble substance.
6. Encapsulated detergent composition according to any of claims 1-5, wherein the capsule is a gelatin capsule.



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# EUROPEAN SEARCH REPORT

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EP 89 20 0931

DOCUMENTS CONSIDERED TO BE RELEVANT			CLASSIFICATION OF THE APPLICATION (Int. Cl. 4)
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	
Y	FR-A-2 143 832 (THE PROCTER & GAMBLE CO.) * claims; page 8, lines 30-40; page 9, lines 1-7; page 10, lines 37-40; page 12, lines 19-40; page 13, lines 1-17 *	1,5,6	C 11 D 17/00
A	---	2,3	
Y	DE-A-2 166 054 (LION FIAT & OIL CO.) * claims; examples 1-4 *	1,5,6	
A	(PATENT ABSTRACTS OF JAPAN) vol. 12, no. 84 (C-482)(2931), 17 March 1988; & JP-A-62223111 (TSUMURA JUNTENDO INC.) 01.10.1987 * abstract *	1,2,5,6	
A	FR-A-2 169 720 (MAJORICA HEUSCH) * claims *	1,5,6	
A	GB-A-1 207 777 (UNILEVER) * claims *	1,5,6	
	-----		TECHNICAL FIELDS SEARCHED (Int. Cl. 4)
			C 11 D
The present search report has been drawn up for all claims			
Place of search	Date of completion of the search	Examiner	
BERLIN	30-07-1990	PELLI-WABLAT B	
CATEGORY OF CITED DOCUMENTS			
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document	